

NACA TN 4259 00901



NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 4259

TEMPERATURE-PRESSURE-TIME RELATIONS IN
A CLOSED CRYOGENIC CONTAINER

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Washington
February 1958

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SUMMARY

Experience has shown that the pressure in a closed cryogenic container may be considerably greater than the vapor pressure that corresponds to the average liquid temperature. An experimental study using liquid nitrogen was made to verify the existence of a temperature gradient as the factor causing the increase in pressure and to investigate the effect on pressure of altering the temperature gradient. The container was provided with means for stirring the liquid and for mixing the vapor. The effect on pressure of different heat-leak rates into the container and different quantities of liquid was also investigated.

The study showed that surface temperature controlled the pressure in a closed cryogenic container and verified the existence of temperature gradients in the liquid, which accounted for the increase in pressure above the vapor pressure corresponding to the average liquid temperature. Stirring the liquid to equalize temperatures caused a reduction in pressure which would extend the time required to reach a limiting pressure. If the design of a closed container will not accommodate a means for stirring the liquid, a lower pressure can be achieved with a given total heat-leak rate by minimizing the direct heat flow to the liquid surface. Even a momentary increase in direct heat flow to the stable liquid layer at the surface should be avoided in order to achieve minimum pressure in a closed container.

INTRODUCTION

One of the problems in the storage of cryogenic liquids is the increase in pressure in the closed container. A considerable quantity of heat may be transferred into the container because of the large difference between the ambient temperature outside the container and the temperature of the cryogenic liquid. The increase in pressure in a closed container due to this heat leak may be obtained from the increase in liquid temperature, calculated from the heat-leak rate, and the vapor-pressure - temperature equilibrium relation. Experience has shown, however, that the

container pressure may be considerably greater than that corresponding to an average liquid temperature. This difference in pressure has been attributed to the development of temperature gradients in the liquid (refs. 1 and 2).

As part of the low-temperature work at the NACA Lewis Laboratory, an experimental study was made to verify the existence of a temperature gradient as the factor causing the difference in pressure and to investigate the effect on pressure of altering the temperature gradient. The container pressure and the liquid and vapor temperatures of liquid nitrogen were measured in a closed cryogenic container. The container was provided with means for stirring the liquid and for mixing the vapor. The effect on pressure of different heat-leak rates into the container and different quantities of liquid was also investigated.

APPARATUS AND PROCEDURE

The liquid nitrogen was contained in a 30- by 93-inch metal Dewar vessel (fig. 1(a)). The Dewar vessel consisted of a 0.037-inch-thick, 304 stainless-steel inner shell surrounded on the sides and bottom by a vacuum jacket. Radiation to the inner shell was reduced by an aluminum-foil wrap. The lower section was further protected by a copper radiation shield attached to the inner shell and also wrapped in aluminum foil. The Dewar vessel was sealed by an uninsulated steel lid. The liquid was stirred with a hand-cranked propeller. An electric fan was used to mix the vapor.

Temperatures were measured about 2 inches radially inward from the inner-shell wall by using two thermocouple rakes. One rake measured temperatures at 8-inch increments along the entire height of the Dewar vessel; the other rake measured temperatures at 1/4-inch increments near the surface of the liquid. Each rake consisted of 30-gage, copper-constantan thermocouple wires inside a 3/4-inch heavy wall, stainless-steel tube. A typical thermocouple rake is shown schematically in figure 1(b). The thermocouple junction was placed 1/4-inch from the tube using a 1/8-inch-diameter, two-hole ceramic tubing held in place with heating element cement. Heat conduction down the thermocouple lead wire to the junction was minimized by exposing a length of lead wire inside the tubing to the liquid. Holes in the tubing, located between the liquid surface and the underside of the lid, allowed the gas to escape so that the liquid could enter the tubing through the open bottom end.

Temperature measurements were made with respect to a liquid-nitrogen bath maintained at atmospheric pressure. The temperature of this liquid-nitrogen bath was used as the reference temperature of the investigation. This reference temperature (about -320.5°F) was obtained from the vapor-pressure - temperature equilibrium relation of liquid nitrogen (ref. 3) and the local atmospheric pressure. Container pressure was measured by

using a differential manometer and a differential pressure transducer, both of which were referenced to atmospheric pressure. The pressure and the temperature histories for each increment of height were obtained for an established heat-leak rate and a given quantity of liquid nitrogen in the Dewar vessel. The heat-leak rate was changed by changing the pressure in the vacuum jacket.

The measured liquid temperature after stirring the liquid was used to obtain the increase in the average liquid temperature. The measured increase in average liquid temperature was divided by the time interval that the container had been closed to obtain the average rate of increase in average liquid temperature ($^{\circ}\text{F/hr}$). This average rate of increase in average liquid temperature was used as an indication of the heat-leak rate into the container for the purpose of this investigation.

The pressure corresponding to the average liquid temperature at any time after the container was closed is defined hereinafter as the calculated pressure. This calculated pressure was obtained from the average liquid temperature using the vapor-pressure - temperature equilibrium relation of liquid nitrogen that was obtained from reference 3 and is shown in figure 2. The average liquid temperature at any arbitrary time interval was obtained from the product of the time interval and the average rate of increase in the average liquid temperature.

The vapor-pressure - temperature curve was also used to determine the saturation liquid temperature corresponding to the measured pressure. This saturation liquid temperature was used to correlate the experimental pressure with the measured liquid temperature.

RESULTS AND DISCUSSION

Typical Pressure History and Temperature Gradient

A comparison of a typical experimental pressure history and the calculated increase in pressure with time is presented in figure 3. The experimental pressure obtained without stirring the liquid or mixing the vapor and the calculated pressure are shown for a heat-leak rate that resulted in a rate of increase in the average liquid temperature of 9.2°F per hour with the container 30 percent full. The experimental pressure exceeded the calculated pressure from the instant the container was closed, and the difference between the two pressures continued to increase with time. The actual pressure developed in two distinct phases: a rapid increase in pressure during the first 2 minutes that the container was closed, followed by a linear increase in pressure with time. These phases of pressure development are accounted for in a later discussion. After 30 minutes, the experimental pressure was about $1\frac{1}{2}$ times the calculated

pressure. Conversely, the calculated pressure was experienced in about one-half the anticipated time.

Temperature gradients measured before and after closing the container are shown in figure 4. These data are for the same conditions of heat-leak rate and liquid content shown previously in figure 3. Temperatures measured at 8-inch increments while the container was vented and after the container had been closed for about 25 minutes are presented in figure 4(a). Temperatures varied from near reference temperature in the liquid at the bottom of the container to near room temperature in the vapor at the top with a sudden increase in temperature near the surface of the liquid.

Temperatures measured at 1/4-inch increments near the surface are shown on an enlarged scale in figure 4(b). Temperatures measured while the container was vented, after the container had been closed for about 2 minutes, and after the container had been closed for about 25 minutes are shown. With the container vented, surface or reference temperature was less than the temperature of the bulk of the liquid. This decrease in temperature at the surface (0.3°F) was in accord with past experience in heat transfer (ref. 4) and is a result of evaporative cooling with flow of heat from the liquid with a free surface. When the container was closed, evaporative cooling at the surface decreased, but the flow of heat to the liquid (due to heat leak) continued and resulted in a reversal of the liquid temperature gradient after the container had been closed for about 2 minutes (fig. 4(b)). This reversal of the liquid temperature gradient probably accounts for the first phase of pressure development (fig. 3). After the container had been closed for about 25 minutes, the average liquid temperature was about 3.8°F above reference, whereas, the liquid temperatures near the surface were more than 4.6°F above reference (fig. 4(b)).

The temperature history of several incremental heights near the surface and at the bottom of the container is presented in figure 5. The experimental pressure data, in terms of the saturation liquid temperature, are also shown. The rapid increase in temperature at a height of 29 inches after the container was first closed was typical of the temperature of the thermocouple probes located in the vapor above this height. While the temperature of the vapor above a height of 29 inches continued to rise, a decrease in temperature was experienced at this height after about 20 minutes. This decrease in temperature was considered to result from liquid wetting the thermocouple. Further consideration of liquid expansion due to the increase in temperature of the liquid at the bottom of the container (height, $2\frac{1}{2}$ in.), when applied to the liquid volume that corresponds to a height of 29 inches, indicated that the liquid level would have raised about $1/2$ inch in a 30-minute time interval. Consequently, liquid surface temperature was considered to lie between the temperature

at a height of $28\frac{1}{2}$ inches and the temperature at a height of 29 inches for the duration of the time interval shown in figure 5. The saturation liquid temperature also lies within the same limits. In other words, the liquid surface temperature controlled the experimental pressure. The temperature of the liquid near the surface (height, $28\frac{1}{2}$ in., fig. 5) increased approximately linearly with time after the first few minutes and probably accounts for the linearity of the second phase of pressure development discussed previously in conjunction with figure 3. The difference between the liquid temperatures at the bottom and at the surface also increased with time, which shows the development of the liquid temperature gradient.

The temperatures near the liquid surface for the other heat-leak rates and liquid contents investigated are presented in figure 6. Similar agreement between liquid surface temperature and saturation liquid temperature (or experimental pressure) was experienced with each heat-leak rate and liquid content investigated.

Effect of Stirring Liquid on Pressure

Because the difference between the actual and the calculated pressures is due to the difference between the surface temperature and the average liquid temperature, altering the liquid temperature gradient should have an effect on container pressure. The effect on pressure of altering the liquid temperature gradient was investigated by stirring the liquid to equalize the liquid temperature.

Figure 7 presents liquid temperature data obtained with stirring for the typical case of heat-leak rate and liquid level previously shown in figures 3 to 5. Liquid temperatures near the surface and at the bottom and the saturation liquid temperature are shown as functions of time, and the time interval when the liquid was being stirred is also indicated. The change in saturation liquid temperature (experimental pressure) with time followed the change in temperatures near the surface (heights of $28\frac{1}{2}$ to 29 in.). Stirring the liquid decreased the temperature near the surface and slightly increased the temperature at the bottom until the liquid temperatures were equalized. The minimum saturation liquid temperature with stirring was obtained shortly after the liquid temperatures were equalized and corresponded within 0.3° F to the average liquid temperature. These data also show that the existence of the liquid temperature gradient caused the difference between the experimental and calculated pressures.

Effect of Heat-Leak Rate and Liquid Content on Pressure

If the liquid is not stirred to equalize temperatures, further consideration of the factors that caused the liquid temperature gradient to develop may be of benefit to the designers of cryogenic containers. The increase in liquid temperature at the surface with the container of this investigation closed was probably due to two factors:

(1) Conduction of heat through the inner shell from the vacuum jacket, which with natural convection caused the heated, less dense liquid to accumulate at the surface

(2) Heat conduction downward from the uninsulated steel lid along the inner shell and also through the quiescent vapor, which brought heat directly to the surface

The relative influence of heat conduction through the vacuum jacket to the liquid or down from the lid directly to the surface was investigated with this container by changing the quantity of liquid. With the same pressure in the vacuum jacket, heat conduction through the inner shell per pound of liquid remains about the same; but with a greater liquid content, the liquid surface is brought closer to the uninsulated lid. The shorter heat path from the lid to the surface, both down the inner shell and through the vapor, should result in greater relative heat flow to the surface. The effect of this increase in relative heat flow to the surface would be to increase the surface temperature and, consequently, increase the pressure.

This higher pressure was experienced as shown by the results presented in figure 8. With a vacuum jacket pressure of less than 5 microns, a higher pressure was obtained with the greater liquid content at any equal interval of time (fig. 8(a)). Increasing the vacuum jacket pressure to about 200 microns with the same liquid content resulted in an increase in container pressure for the same time interval because of an increase in heat conduction per pound of liquid through the inner shell; but a further increase in pressure was obtained when the liquid content was increased (fig. 8(b)). These data show that in order to minimize the pressure developed without stirring the liquid, consideration should be given not only to reducing the total heat-leak rate but also to minimizing the direct heat flow to the surface.

Effect of Vapor Mixing on Pressure

Another factor that may affect the flow of heat to the surface is the temperature gradient of the vapor. Natural convection in the vapor causes the heated, less dense vapor to accumulate at the top of the container. The warmer vapor, which is farthest from the surface, is

beneficial because less conduction takes place between the cooler vapor near the surface and the liquid layer at the surface. Altering the temperature gradient of the vapor by forced convection may result in more heat accumulating at the surface, with a resultant increase in surface temperature and a subsequent increase in pressure. The effect on pressure of altering the temperature gradient of the vapor was investigated by using an electric fan to mix the vapor. The fan was installed in a fixed position, which required that the liquid content be changed in order to vary the height of the fan above the surface.

The pressures obtained with vapor mixing for two liquid contents are shown in figure 9(a) for a vacuum-jacket pressure of less than 5 microns. The normal increase in pressure with time without vapor mixing is shown with the container 70 percent full in the left side of figure 9(a). A substantial increase in pressure occurred during the 1-minute interval when the fan was operating. Stopping the fan caused a slight decrease in pressure, but the pressure then remained steady for about an 8-minute interval, with no indication of a further decrease. The fan was 7 inches above the surface in this case. Increasing the height of the fan above the surface to 44 inches by decreasing the liquid content also resulted in an increase in pressure with mixing, but of considerably less magnitude. Similar results were obtained with vapor mixing when the vacuum-jacket pressure was increased to about 200 microns (fig. 9(b)). In this case, the fan was either 17 or 45 inches above the surface. The difference in the pressures resulting from vapor mixing is attributed to the difference in the movement of the vapor above the surface. These results show that, in order to maintain minimum pressure in a closed cryogenic container, even a momentary increase in direct heat flow to the stable liquid layer at the surface should be avoided.

SUMMARY OF RESULTS

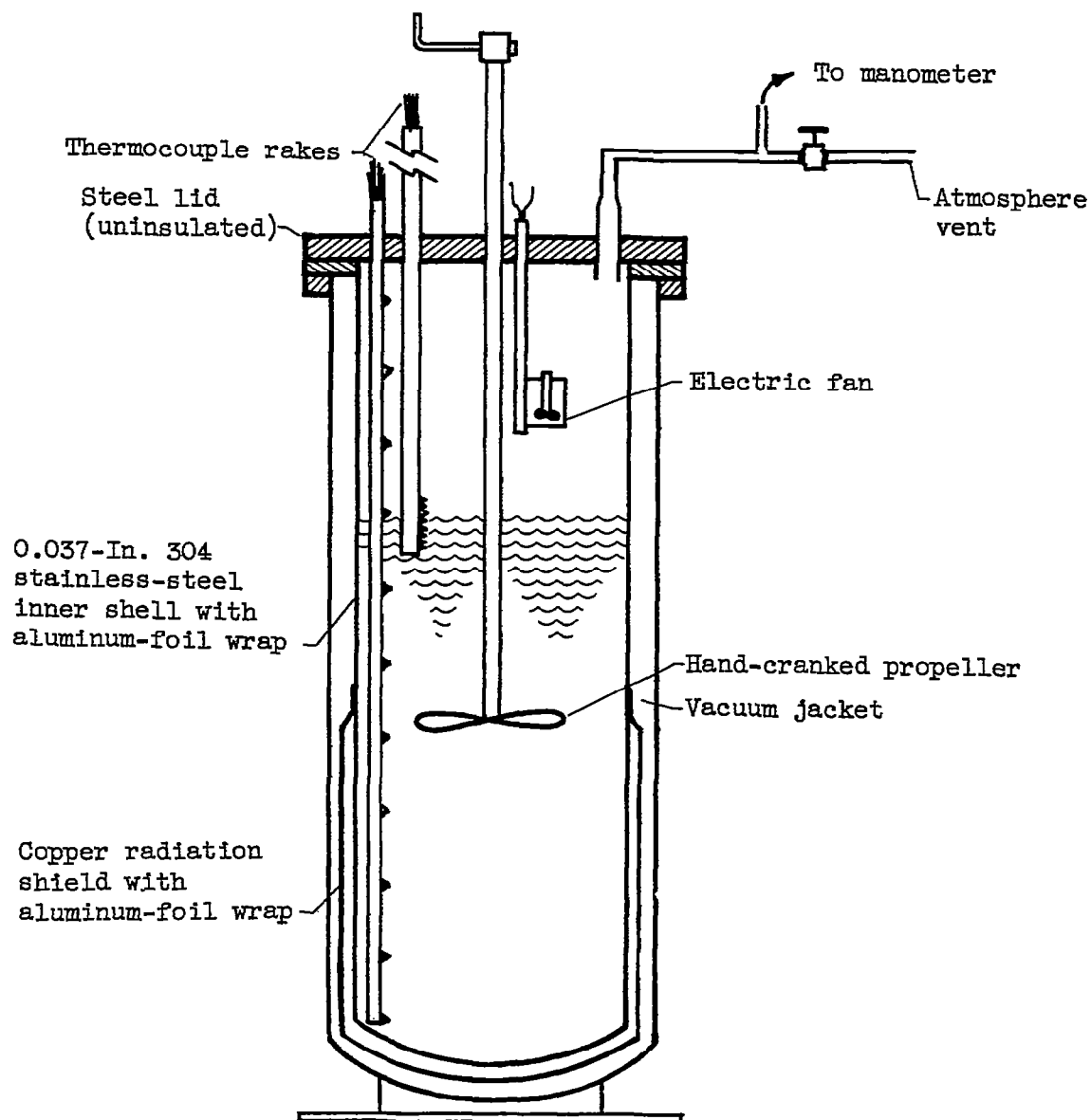
This experimental study with liquid nitrogen has shown that surface temperature controlled the pressure in a closed cryogenic container and verified the existence of temperature gradients in the liquid. The gradients became more severe with time and accounted for the increase in pressure above that calculated from the vapor-pressure - temperature equilibrium relation based on the average liquid temperature. Stirring the liquid to equalize liquid temperatures caused a reduction in pressure, which would extend the time required to reach a limiting pressure. If the design of a closed container will not accommodate the stirring of the liquid, a lower pressure can be achieved with a given total heat-leak rate by minimizing the direct heat flow to the liquid surface. Even a momentary

increase in heat flow directly to the stable liquid layer at the surface should be avoided to achieve minimum pressure in a closed container.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 17, 1957

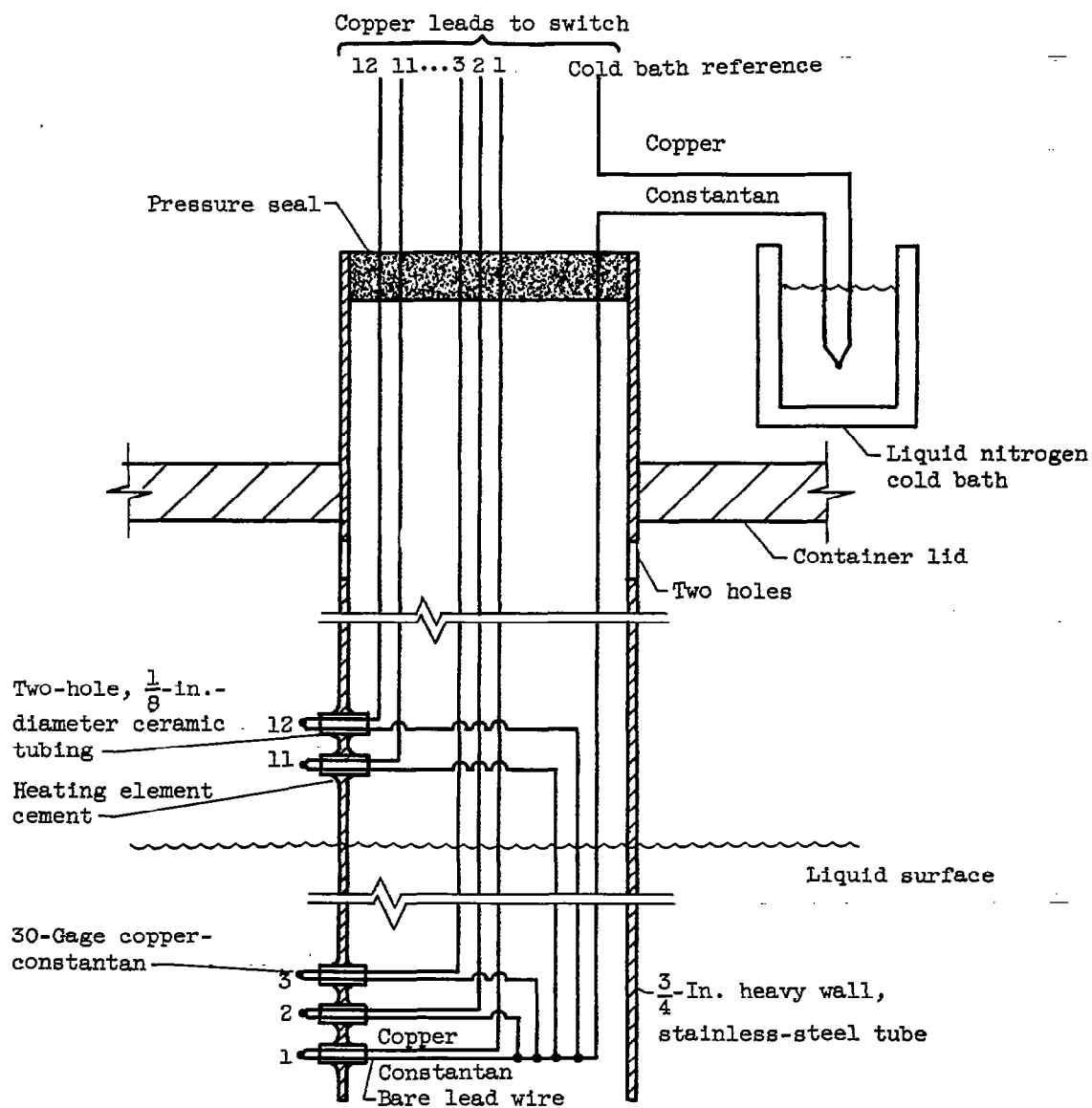
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3. Hilsenrath, Joseph, et al: Tables of Thermal Properties of Gases. Cir. 564, NBS, Nov. 1, 1955.
4. Jakob, Max: Heat Transfer. Vol. I. John Wiley & Sons, Inc., 1949, p. 618.



(a) 30 By 93-inch metal Dewar vessel.

Figure 1. - Test apparatus.



(b) Typical thermocouple rake.

Figure 1. - Concluded. Test apparatus.

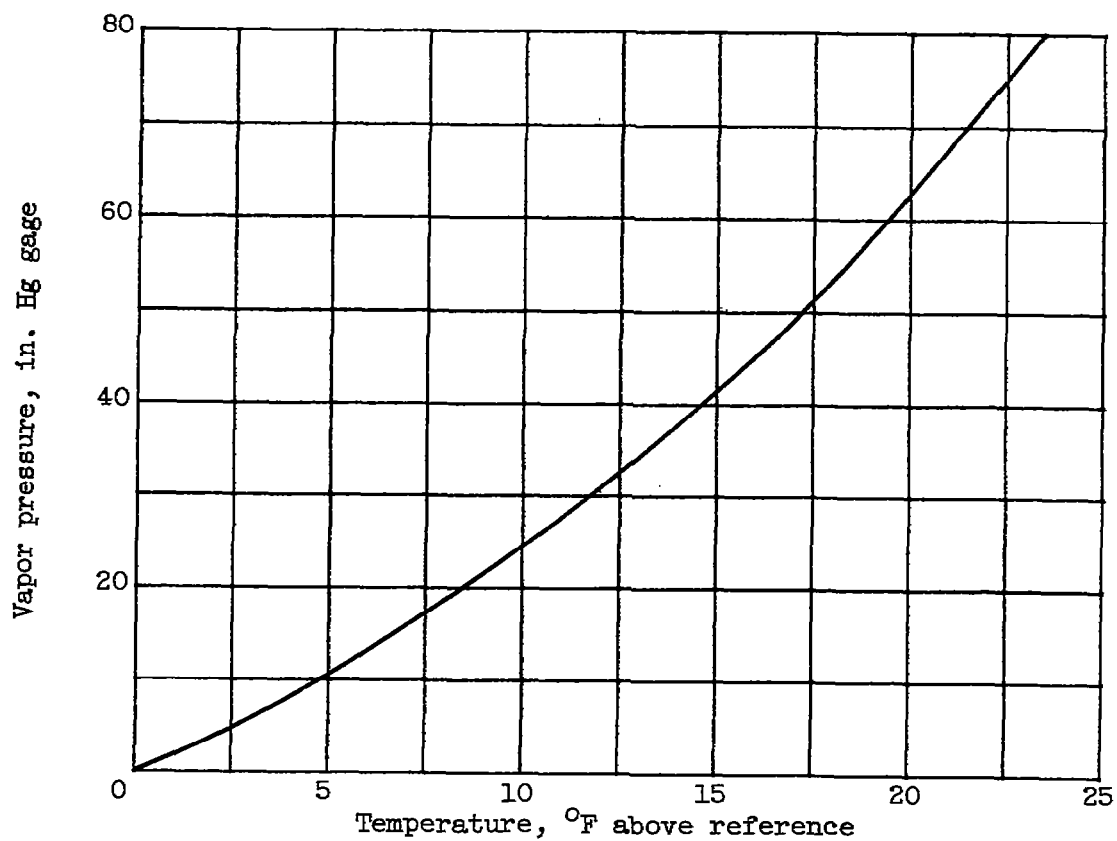


Figure 2. - Vapor-pressure - temperature equilibrium relation of liquid nitrogen (ref. 3). Atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.

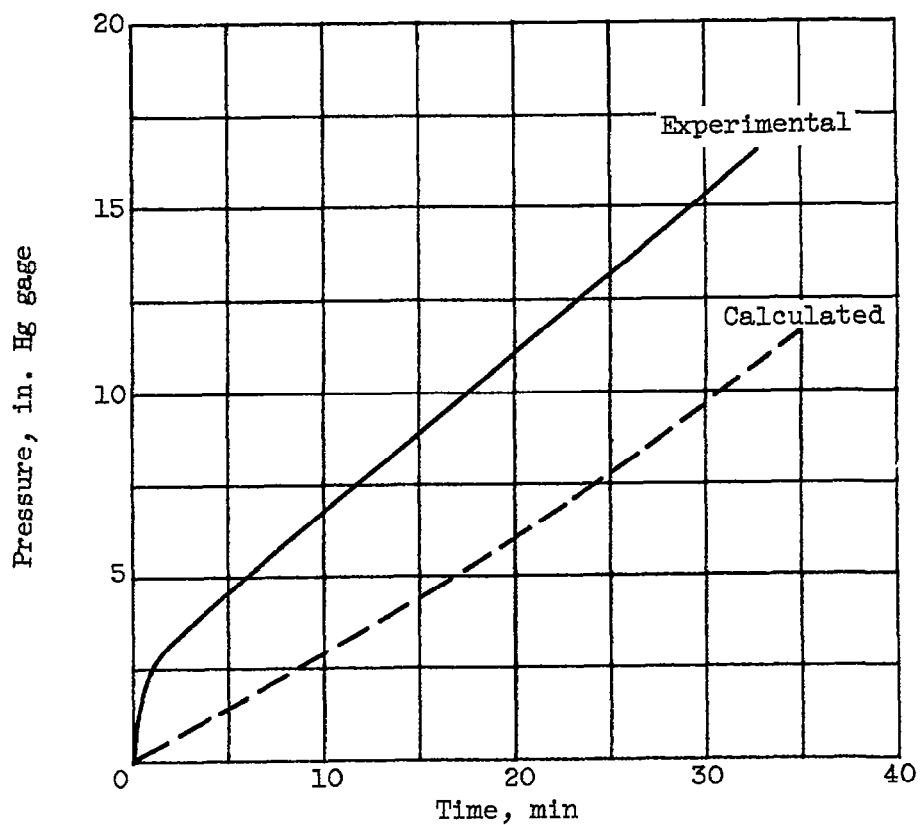
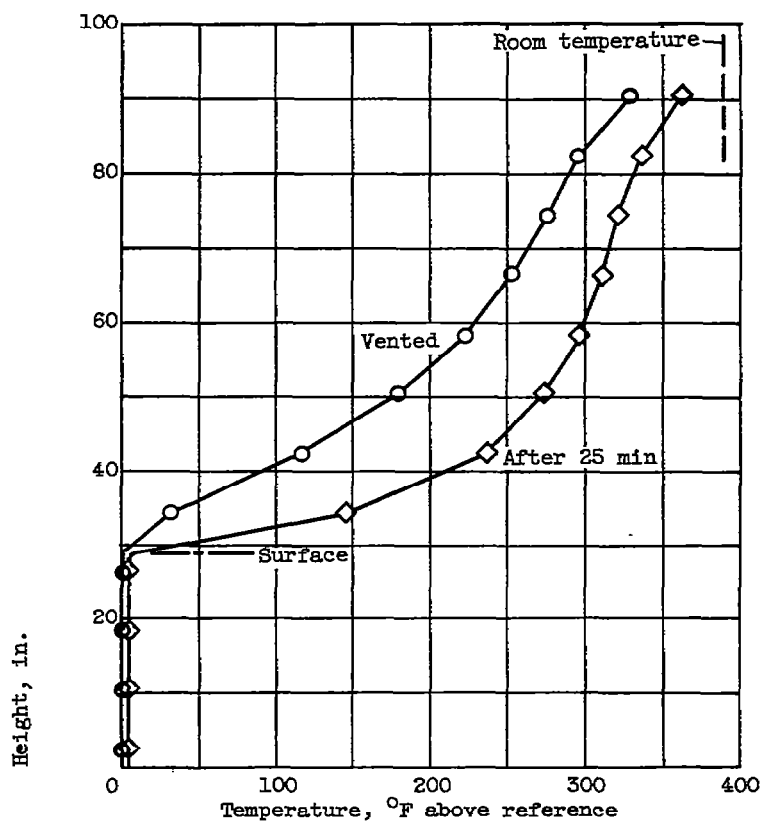
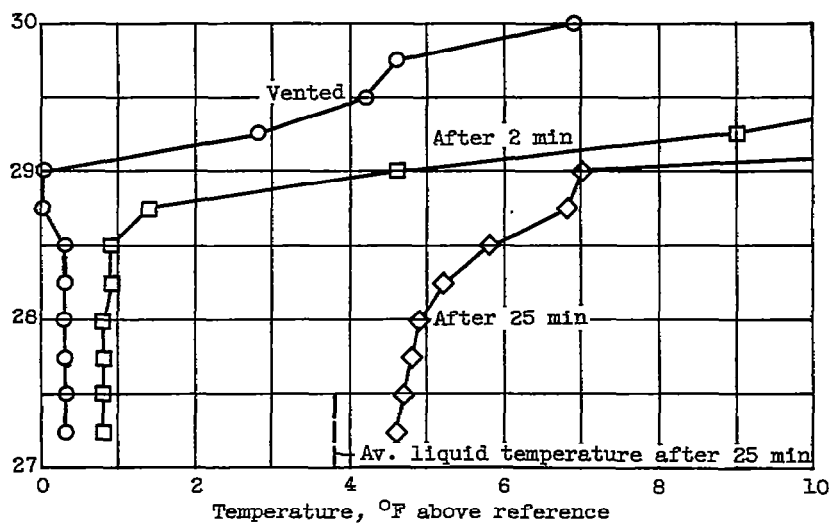


Figure 3. - Typical pressure history. Heat-leak rate, 9.2° F per hour; liquid content, 30 percent; atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.



(a) Temperatures at 8-inch increments.



(b) Temperatures at 1/4-inch increments.

Figure 4. - Typical temperature gradients. Heat-leak rate, 9.2° F per hour; liquid content, 30 percent; atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.

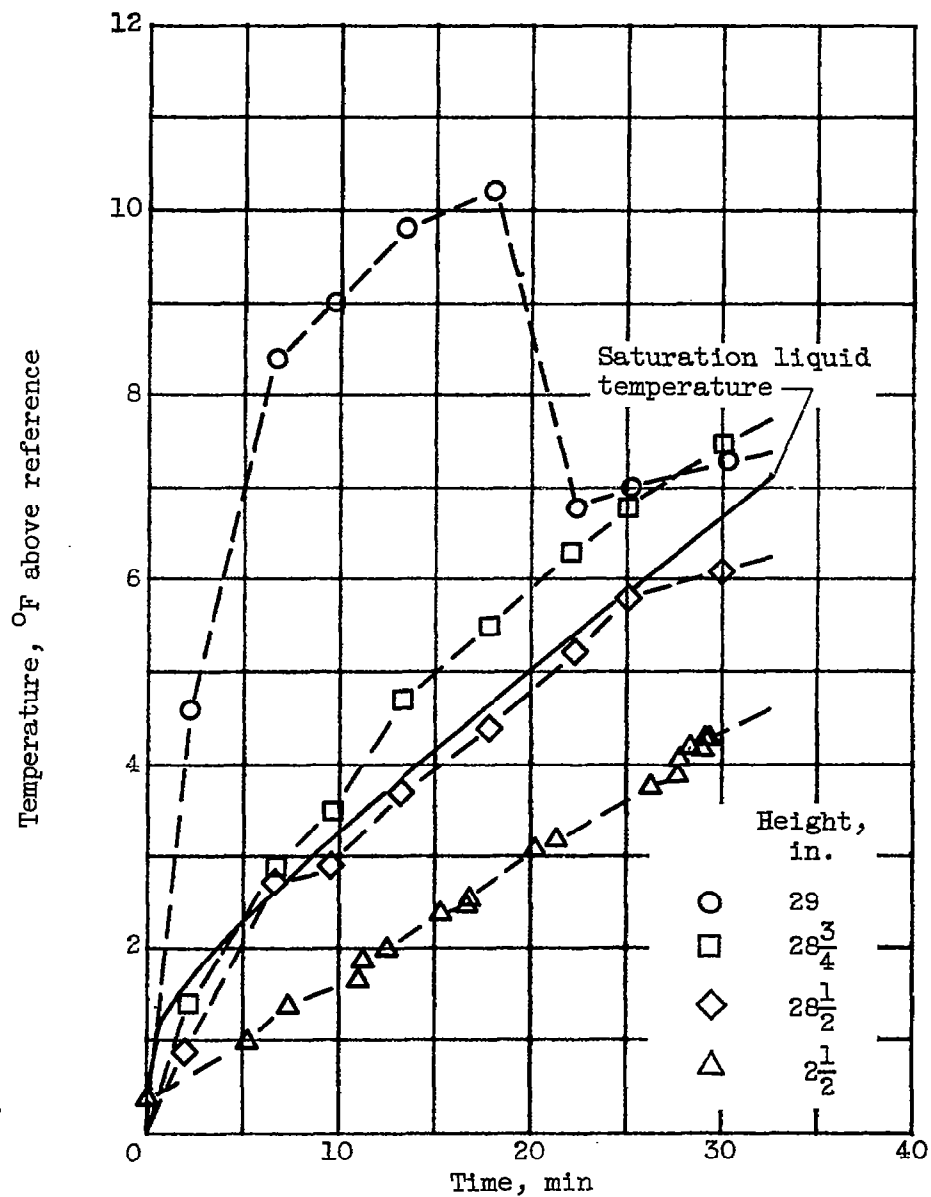
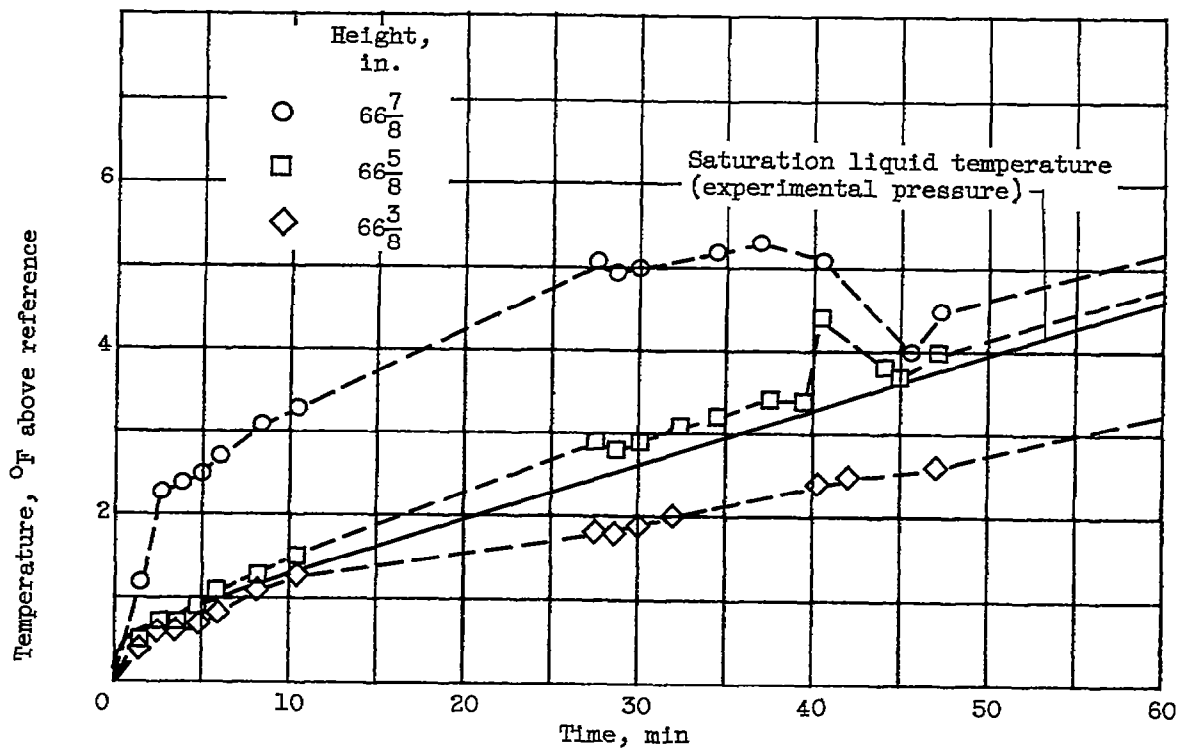
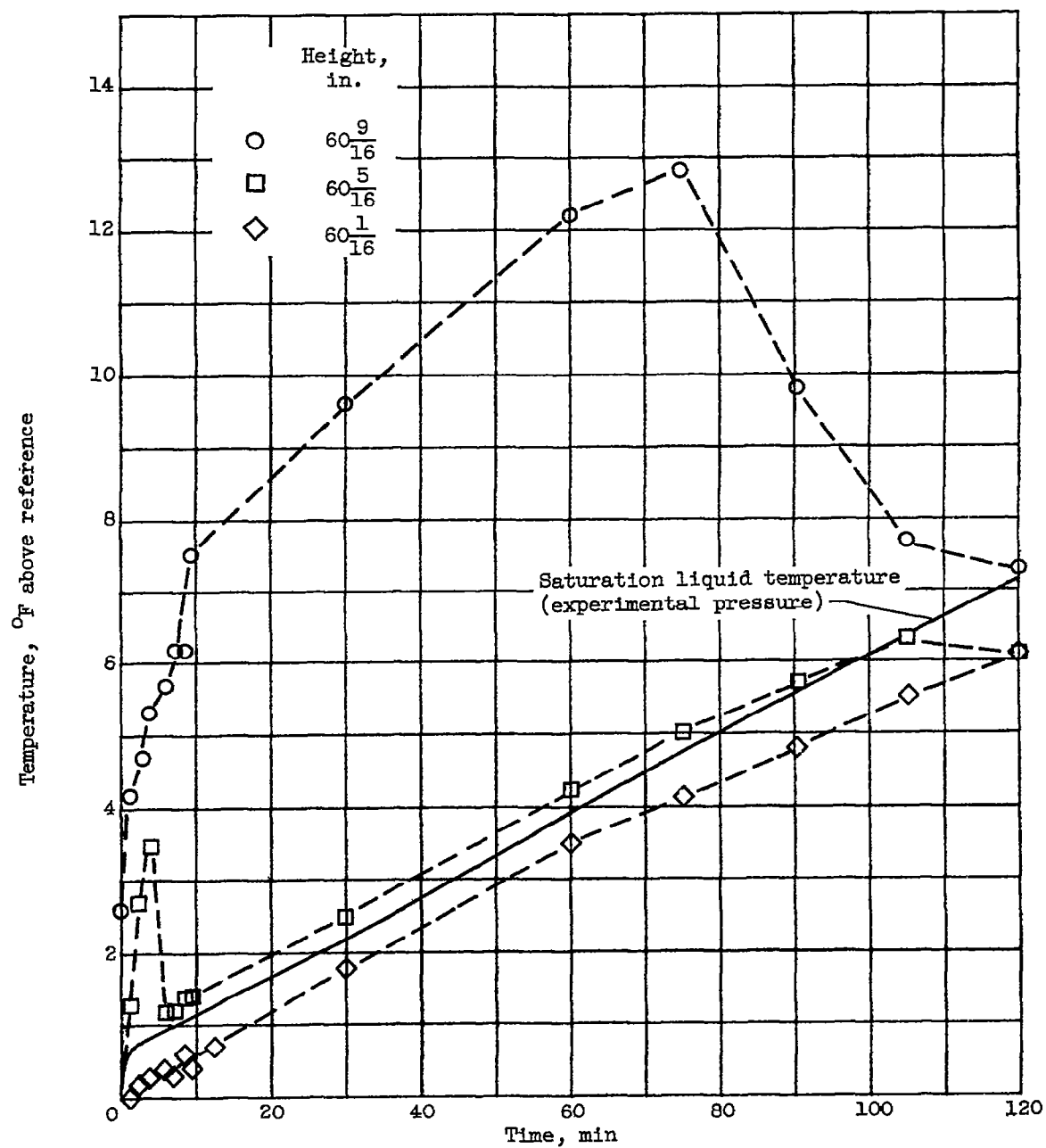


Figure 5. - Typical temperature history. Heat-leak rate, 9.2°F per hour; liquid content, 30 percent; atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5°F .



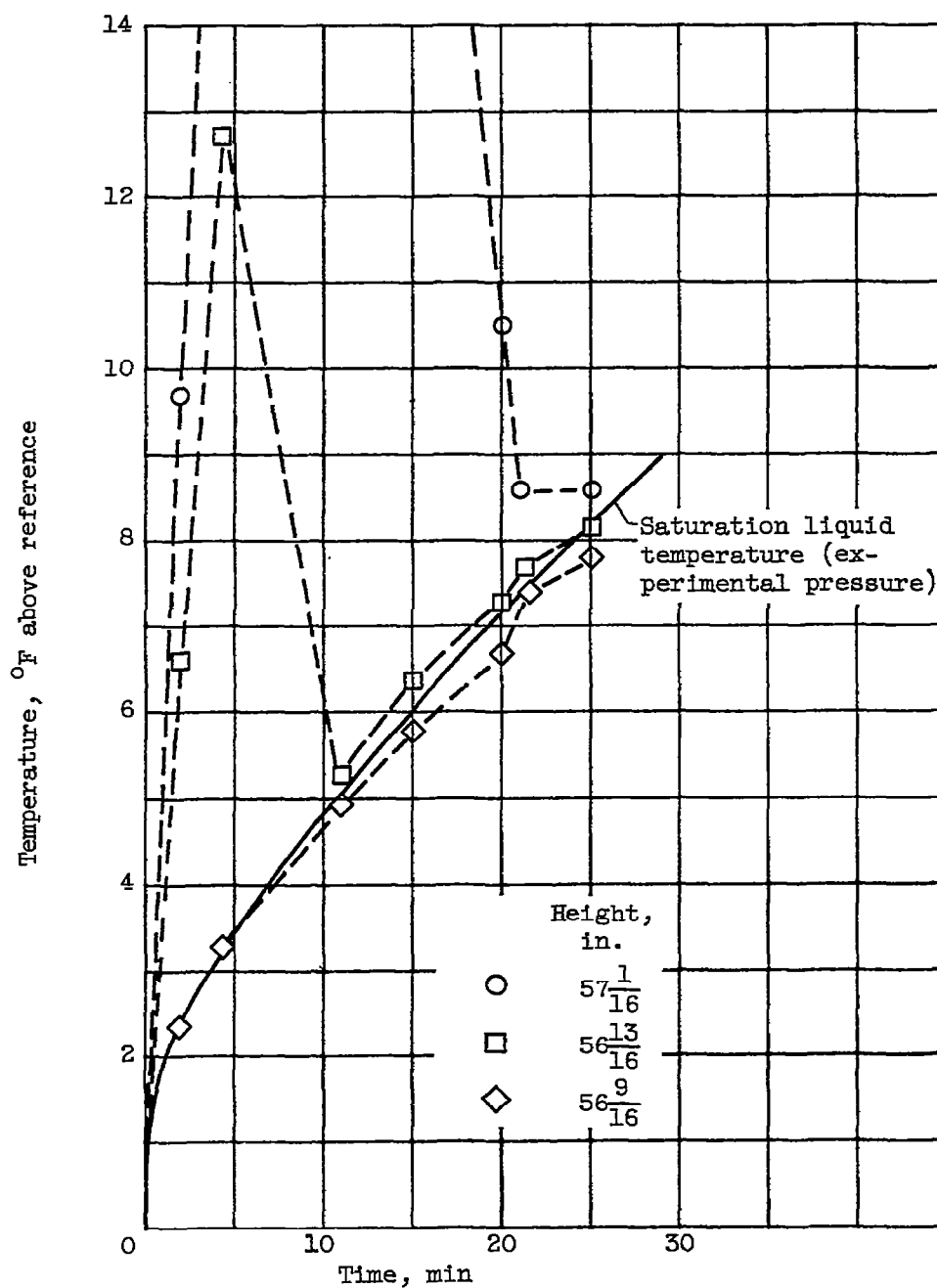
(a) Heat-leak rate, $< 1^{\circ}$ F per hour; liquid content, 70 percent.

Figure 6. - Temperatures near liquid surface. Atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.



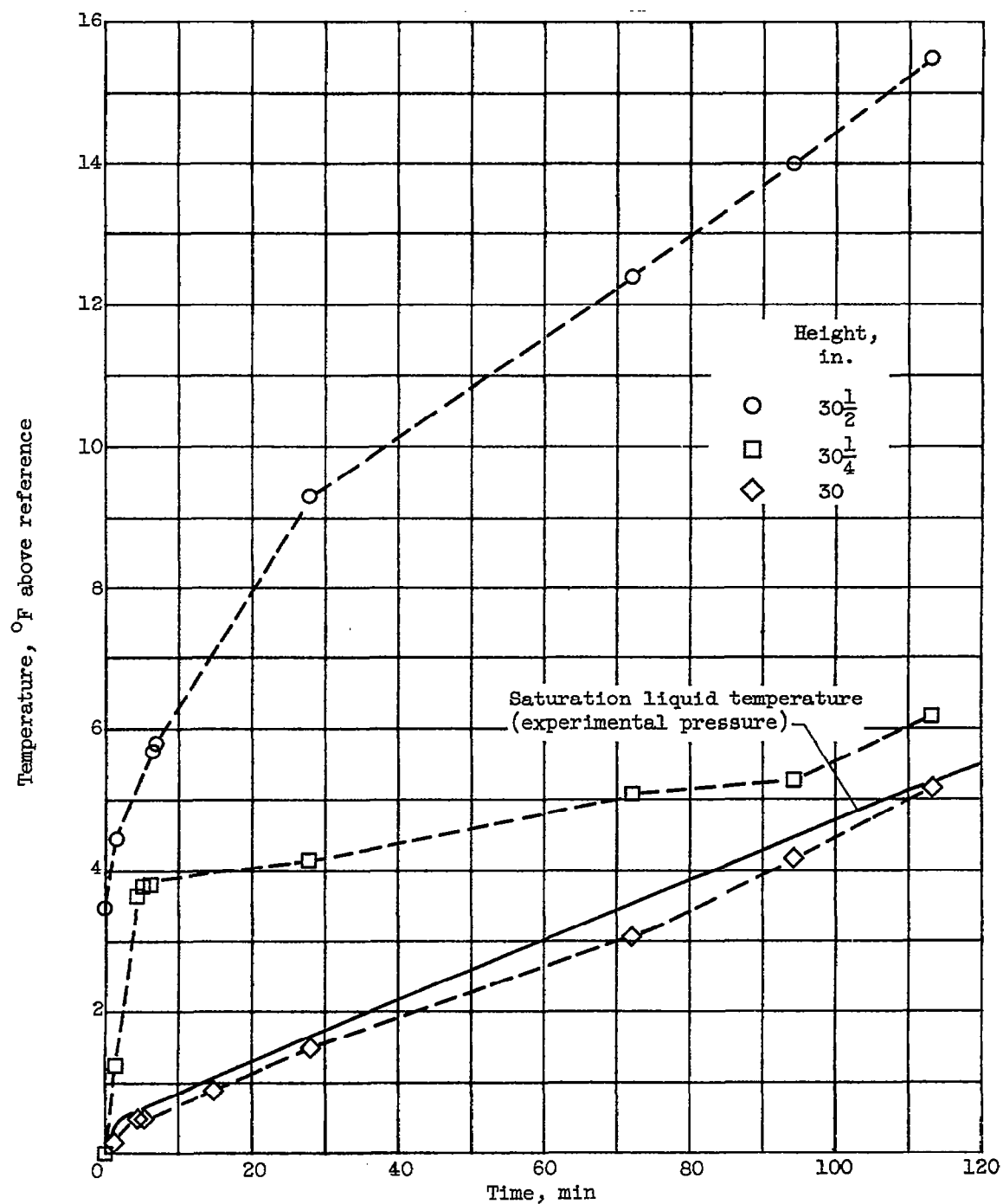
(b) Heat-leak rate, 1.2° F per hour; liquid content, 60 percent.

Figure 6. - Continued. Temperatures near liquid surface. Atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.



(c) Heat-leak rate, 10.4° F per hour;
liquid content, 60 percent.

Figure 6. - Continued. Temperatures near liquid surface. Atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.



(d) Heat-leak rate, 1.1° F per hour; liquid content, 30 percent.

Figure 6. - Concluded. Temperatures near liquid surface. Atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.

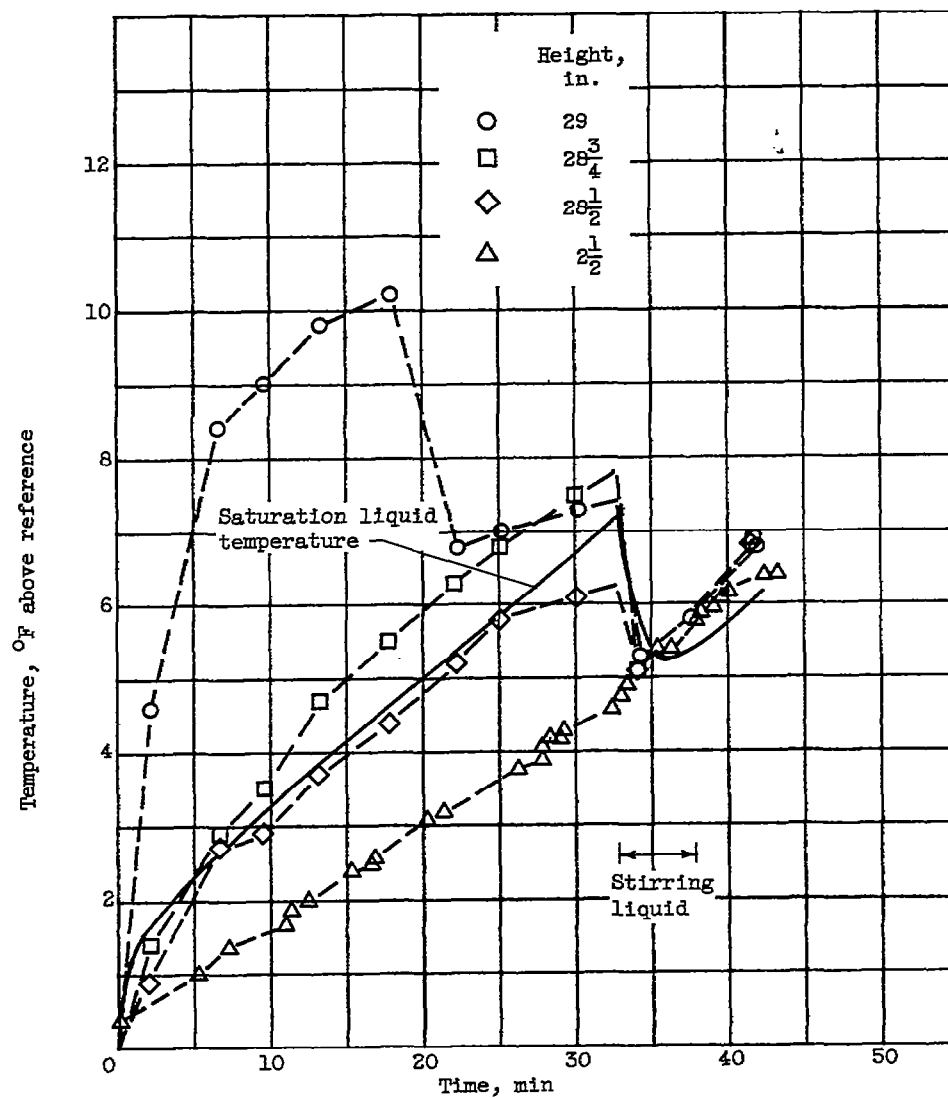
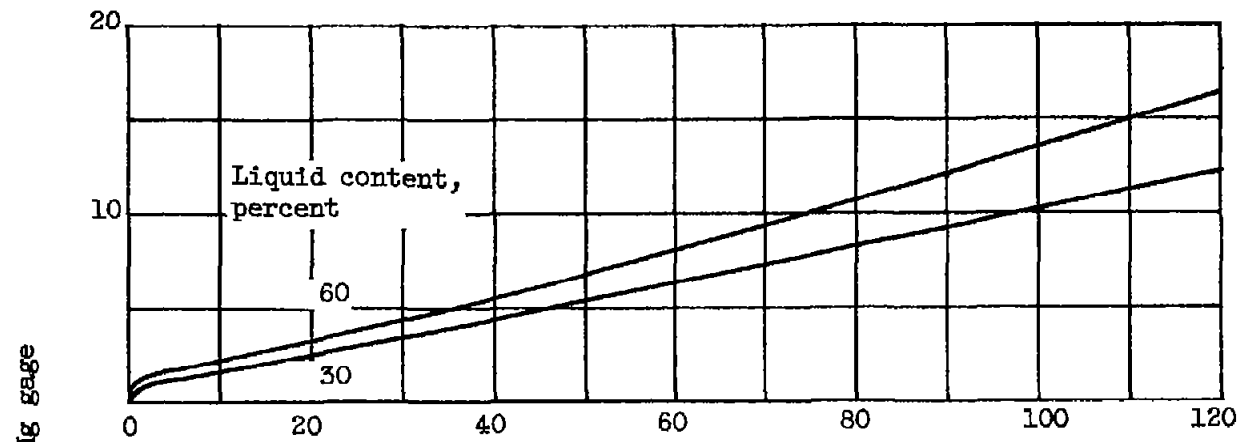
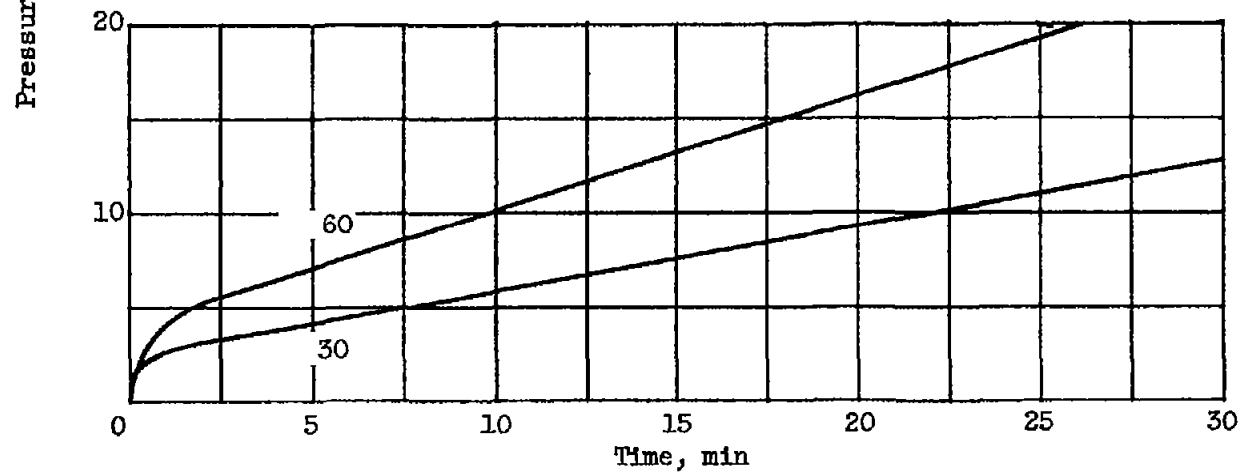


Figure 7. - Temperature data when stirring liquid. Heat-leak rate, 9.2° F per hour; liquid content, 30 percent; atmospheric pressure, 29.7 inches of mercury absolute; reference temperature, -320.5° F.



(a) Vacuum jacket pressure, <5 microns.



(b) Vacuum jacket pressure, ≈200 microns.

Figure 8. - Effect of heat-leak rate and liquid content on pressure.

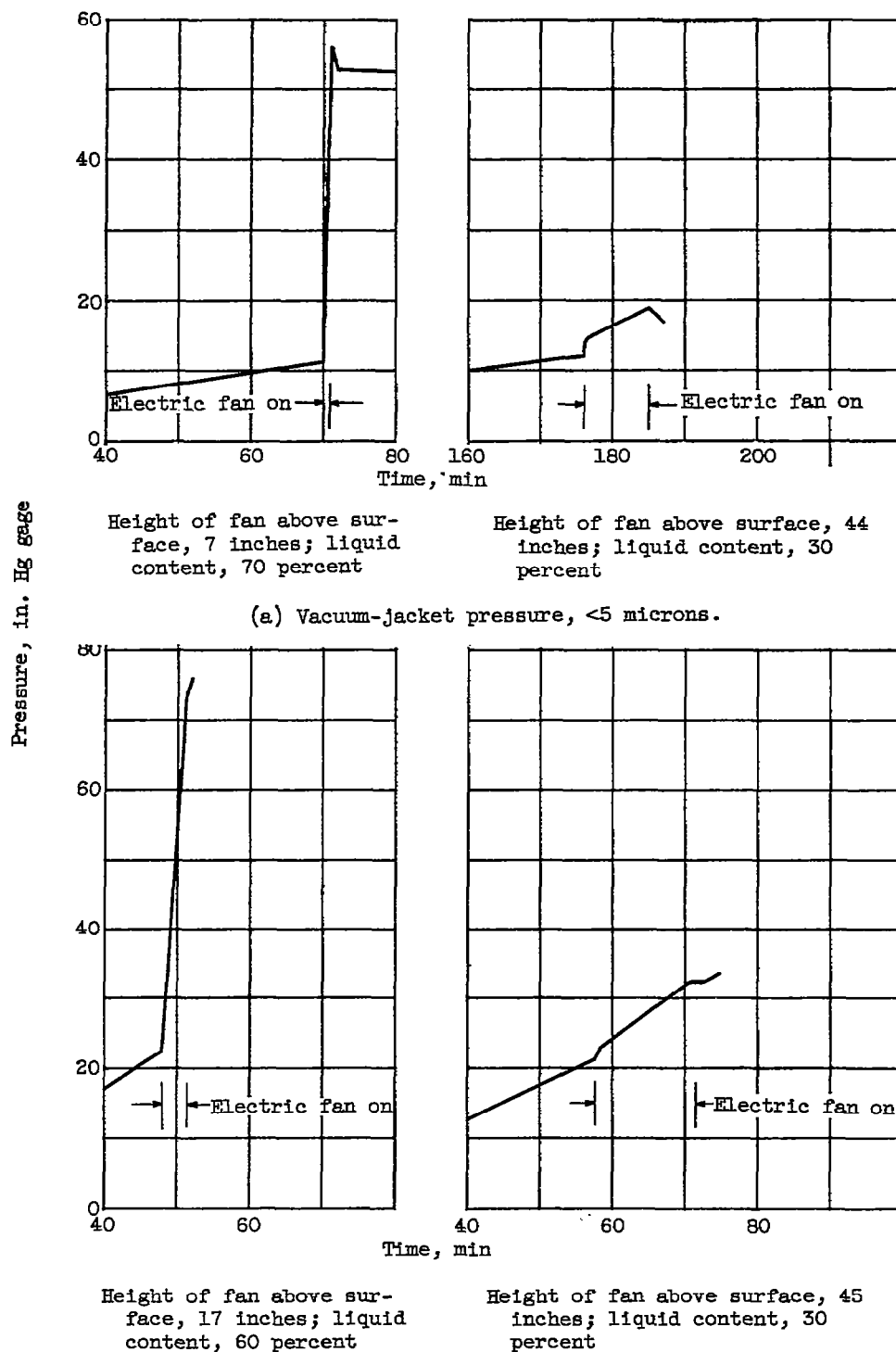
(b) Vacuum-jacket pressure, ≈ 200 microns.

Figure 9. - Pressure data with vapor mixing.